

XXI. *Experiments and Observations to investigate the Composition of James's Powder.* By George Pearson, M. D. F. R. S.; communicated by Sir Joseph Banks, Bart. P. R. S.

Read June 23, 1791.

THE medicine upon which many physicians principally depend in the cure of continued fevers is JAMES's *Powder*; but, although it has been very extensively used above thirty years, the public have not, I believe, been informed of the particular nature of this substance. This *powder* was originally a patent medicine; but it is well known that it cannot be prepared by following the directions of the specification in the Court of Chancery. Presuming that I have made some experiments and observations which may explain the nature and manner of preparing this medicine, and, perhaps, may extend the history of antimony; I beg leave to have the honour of presenting an account of them to the Royal Society.

*Sensible properties of JAMES's Powder.*

Some parcels of this preparation are white, but in general it has a yellowish cast; and this shade is more evident in some specimens than in others. It is said, that this powder cannot, in general, be made at different times of precisely the same shade of yellow or degree of whiteness. Sometimes with the aid of a lens a few very small shining *spicula* are seen mixed with powder. When pressed between the fingers it feels  
smooth,

smooth, with some rather rough particles, and it is gritty in the mouth. Most parcels at first are tasteless; but in about a minute there is a slight brassy taste. It is perfectly inodorous.

*Specific gravity.*

This powder feels much heavier than any of the common earths and stones in a pulverized state. One of the phials, nearly full, in which it is sold, reckoned to hold a quantity equal to twelve packets, or 480 grains, contained 470 grains troy weight of JAMES's powder. This phial, filled with distilled water to the same height that it had been by the powder, was found to contain nearly four drachm-measures, or about 240 grains, of this liquid \*.

*Effects of fire.*

(a) The exterior part of the flame of a candle applied, by means of the blow-pipe, to about one, two, and three grains of JAMES's Powder on charcoal, and also in the spoon, only made it yellowish while hot, but, on cooling, this colour disappeared. The interior and hottest part of the blue flame turned this powder yellow, and when continued so as to ignite it, a white inodorous fume or vapour arose, which soon ceased; and though the heat was continued, the powder neither appeared to diminish nor melt; but, on cooling, a slightly co-

\* After this Paper had been read, an experiment, in a different manner, was made to ascertain the specific gravity of this powder. The quantity which nearly filled a phial weighed 437 grains; and filling the same phial, to the same height, with distilled water, the temperature of which was 65°, the water weighed 250,2 grains. The reason of the variation in these results, in making use of different parcels of this medicine, will be obvious, from the following account of its preparation, and the great difficulty of determining, with accuracy, the specific gravity of powders.

hering white solid, about three-fourths of the original weight of the powder, was left. If the flame was suddenly withdrawn, as soon as the white fumes appeared, they ascended with a kind of revolving motion.

(*b*) Two grains of this powder, mixed with about three of pulverized tartar, being exposed on charcoal to flame applied by the blow-pipe, the mixture turned black, boiled, and swelled; and by continuing to apply the flame, the coaly matter of the tartar disappeared, a part of the mixture fused, and in that state several small, silvery, apparently metallic grains were perceived. On cooling, they were seen with the naked eye, or with a lens, adhering to an irregularly figured, partially melted, whitish mass. On a second application of the flame, these metallic globules disappeared.

(*c*) JAMES's Powder, with glass of phosphoric acid, melted into an opaque yellowish globule while hot, which on cooling grew whitish.

(*d*) This Powder, with several times its weight of melted borax, afforded a colourless transparent glass while fluid; but, on adding a larger proportion of powder, the globule turned opaque, and when cold became of a milky whiteness. As the JAMES's Powder mixed, or melted, with the fused salt, slight explosions were several times heard.

(*e*) With fossil alkali, in the spoon, this powder apparently fused, and afforded a colourless transparent fluid in a state of rotatory motion; but on cooling it grew opaque, and had a horny appearance.

(*f*) 100 grains of this powder in a two-inch English crucible, the cover of which was luted on it as closely as possible, were exposed above two hours to a fierce fire in a melting furnace. On breaking this crucible, when cold, the powder was

found changed into an entire very hard white solid, receiving its figure from the vessel, and weighed 95 grains. On breaking this hard solid, the lower part of it seemed to be vitrified, or in a state of enamel; and being powdered, it afforded a much whiter powder, and of greater specific gravity, than before. The degree of fire denoted by WEDGWOOD's pyrometer was 166°.

These experiments indicated the presence of a metallic calx, a part at least of which was that of antimony, mixed with earthy matter.

*Experiments with different menstrua applied to JAMES's Powder.*

I. *With water.*

300 grains of this powder were digested for several hours in twenty-seven ounces of distilled water, and then boiled for one hour. While boiling the water appeared milky; but in half a minute's time, after withdrawing the lamp over which it boiled, the liquid became nearly clear, and the sediment deposited was apparently the powder undiminished in quantity, and in other respects unaltered. While hot the liquid was decanted upon a filter of several folds of paper previously weighed, through which twenty ounces of quite clear liquid, like water, readily passed. Very little powder could be perceived on the filter; but when it was well dried, it weighed fourteen grains more than before the experiment. The filtered liquor was tasteless. In about three quarters of an hour it grew slightly *turbid*, and in ten minutes after became *milky*. On standing eight days longer, the milkiness diminished, and a small quantity, perhaps four grains, of close white sediment, firmly adhering to the sides of the vessel, were deposited. A little of this slightly milky fluid being made hot, it grew quite clear, and on cooling

turned milky as before, or deposited a sediment; therefore, this milkiness depended on cold water dissolving a smaller proportion of JAMES's Powder than hot water. The whole of the filtered milky liquor was poured upon a filter as before, through which it passed without any diminution of its turbid appearance; and, on drying, the filter was found to have increased only a quarter of a grain in weight. Some of the vapour that arose during the ebullition being condensed was found to be pure water.

On a repetition of this experiment the phenomena above related were always observed; but the semi-vitrified JAMES's Powder above-mentioned (*f*) afforded a much less milky fluid and sediment than the powder used in the preceding experiment.

In order to determine the kind of substances in water after boiling in it JAMES's Powder, the following re-agents were added to the above filtered liquor.

1. Acid of sugar sometimes occasioned immediately more turbidness, and at other times transparency was instantly produced; but in all cases, on standing, more sediment fell than from the filtered liquor alone.

2. Muriated barytes in about an hour rendered this liquid evidently more turbid, and on standing more sediment was deposited than from the filtered liquid to which nothing had been added.

3. Lime-water occasioned immediately a curdy appearance.

4. Infusion of turnsole was sometimes turned to a slight red; but in general it was not altered in colour.

5. Nitre of silver produced in a few hours a slight sediment.

6. Prussianated alkali of tartar occasioned no alteration immediately, nor for four days after adding acetous acid to this mixture.

7. Mild alkali of tartar, and likewise mild fossil alkali, though boiled in this liquor, did not occasion any additional precipitation.

The sediment that fell, on merely standing, from the above filtered liquid, was next examined.

1. It did not dissolve in 100 times its weight of boiling hot concentrated acetous acid; nor,

2. Was it apparently acted upon by boiling with mild alkali of tartar; for, after decanting the clear liquid of this mixture, the sediment from it was not dissolved by a large quantity of acetous acid, but readily by marine acid.

3. This sediment immediately disappeared on pouring upon it a much smaller quantity of marine acid, and also of acid of nitre, than had been applied of acetous acid, without any solution ensuing.

4. To one portion of this solution in marine acid just mentioned, was added gradually lixivium of alkali of tartar; after the effervescence had ceased to be produced, the first drop occasioned a turbid appearance, and the liquid did not grow clear again on adding a large proportion of vinegar to make it four.

5. Another portion of this solution in marine acid, being boiled to carry off the superabundant acid, was poured into a large proportion of lime-water, by which it was rendered cloudy, and did not become clear again on adding concentrated acetous acid to make it four.

6. To a third portion of this solution in marine acid, from which the redundant acid had been carried off, twenty drops of Prussian alkaline lixivium were added, which immediately turned it of a bluish cast without disturbing its transparency; and, after standing four days, a smaller quantity of pale blue sediment

sediment was deposited than had fallen from a quantity of distilled water equal to this muriatic solution, to which had been added twenty drops of this Prussian alkaline lixivium, and one-fourth part of a drop of muriated antimony. The same Prussian alkali mixed with vinegar, on standing, turned bluish, but deposited nothing.

7. Though the sediment that fell in the filtered fluid, p. 320. on merely standing, dissolved in the nitrous acid as above mentioned; yet, when this acid was added in a smaller proportion, but to render the mixture sour, a partial solution only took place. On adding, however, a very small quantity of marine acid, the solution was total, and with less superabundance of this menstruum than of the nitrous acid in which a part remained undissolved. This solution

(a) With water deposited Algaroth powder:

(b) With Prussian alkali turned bluish, and, on standing, a small quantity of sediment took place.

(c) A bright plate of copper, immersed in this solution, did not appear to be at all whitened, or rendered paler.

(d) Muriated barytes rendered this solution very turbid instantly.

(e) Acid of sugar produced no change, except in two or three days a very minute portion of sediment; but the saccharated soda immediately occasioned a slight precipitation.

(f) Alkali of tartar, fully aerated, rendered this solution slightly turbid; but caustic mineral alkali induced no change.

(g) Lime-water, in a small proportion, did not affect the transparency; but in a larger produced copious clouds.

The JAMES's Powder which afforded the solution in water, on which the experiments above related were made, was boiled a second time in eighteen ounces of distilled water for

two hours. The decanted and filtered liquid on cooling grew less turbid than before, p. 320. and deposited less sediment. The filter, on drying, was found to have gained ten grains, though very little powder could be seen upon it. The experiments above related, were made on this filtered liquor, and in a flighter degree the same appearances were observed.

The JAMES's Powder remaining after these experiments, being well dried, weighed 260 grains, and therefore was found to have lost 40 grains, partly by solution in water, but still more by its adhering to the filters.

I wished to know the proportion in which JAMES's Powder dissolved in water; and therefore evaporated, in a very thin light glass pan, previously weighed, twenty-four ounces of the filtered liquids, p. 320. and p. 324. Very little precipitated matter appeared till nearly the whole of the liquor was evaporated; and, when the whole was carried off, a tasteless, whitish, leafy, or mica-like sediment, but in some parts black, was left, that weighed six grains. This sediment required above 100 times its weight of hot water to dissolve it. It was boiled in 500 times its weight of distilled water, and it passed through several folds of filtering paper rather turbid even while hot; nor could it be rendered clear by repeated filtration through paper. This filtered liquid,

(a) With infusion of turnsole and turmeric, betrayed no alkaline substance, nor decisively any acidity.

(b) Lime-water rendered it curdy; and on adding vinegar, it grew milky.

(c) With acid of sugar it grew clear; but, on standing, was more turbid than before.

(d) Salited



(d) Salited barytes made it instantly turbid.

(e) Alkalies, mild and caustic, induced no change.

(f) Prussian alkali produced only a clear greenish colour, after the addition of vinegar, and long standing.

(g) A very small quantity of marine acid rendered it quite clear; but it required much more nitrous acid to produce this effect; and this mixture did not whiten copper.

(b) With nitre of silver the filtered liquor turned of a somewhat bluish hue, and afterwards curdy.

The undissolved matter that remained on the filter, p. 324. 1. 22. above mentioned, appeared, on examination, to be the same kind of substance, with a larger proportion of iron, as that which was dissolved by water, the experiments on which have been just mentioned. In particular, it afforded Algaroth powder, but did not whiten, in the smallest degree, a copper plate.

The following conclusions may, perhaps, be justifiably drawn from these experiments on JAMES's Powder with water.

1. That the whole, or a part, is soluble, or at least may be suspended, in about 2000 times its weight of pure water cold; and in about half this quantity of boiling water.

2. That this solution contains calcareous earth united to an acid, or some other substance, from which it cannot be dis-united by caustic or mild fixed alkalies; therefore, the precipitation by muriated barytes cannot be referred to vitriolated lime.

3. That this solution contains a metallic calx, a part of which at least is that of antimony uncombined, or at least not united to any acid with which it forms a compound soluble in water.

4. That

4. That the substance in the nitrous solution of the part of JAMES'S Powder that had been dissolved in water, which precipitates lime from lime-water, and which precipitate is not soluble in a large quantity of vinegar, is, probably, phosphoric acid from phosphorated lime decomposed by nitrous acid.

The precipitation by muriated barytes and nitrated silver could not be from vitriolic and marine acids consistently with the preceding experiments; and I could not have conjectured what was the ingredient in JAMES'S Powder which occasions it, if I had not found, that muriated barytes is not only a test of vitriolic but of phosphoric acid united to lime and alkalies; and the acid of phosphorus will also produce a turbid appearance with nitrated silver. The calx of iron, in the above experiments, is in perhaps too small a quantity to be considered in any other light than as an accidental substance.

## II. *With acetous acid.*

The 260 grains of JAMES'S Powder, remaining after boiling 300 grains of it in distilled water, and after the unavoidable waste of it in the above experiments with water, were put into a tubulated retort that would contain four ounce-measures, on which were poured three ounces of concentrated acetous acid, the specific gravity of which was as 106 to 100, the neck of the retort being immersed in quicksilver, and the tubulated part being immediately closely stopped. No effervescence was perceived; nor did any elastic fluid rise during twenty-four hours into an inverted vessel of quicksilver; and when heat was applied to boil the acid, nothing but the common air of the retort and a little water and acetous acid came over.

This mixture of acetous acid and powder being poured, while hot, on a filter of two folds of paper, a clear and colourless liquid passed through, that remained so, when cold, without any sediment. The filter, with the powder upon it, being well dried, weighed ten grains more than the sum of their weight separately before the experiment; but the powder being carefully scraped off weighed only a little more than 240 grains, and appeared to have suffered no change in its properties.

This filtered liquor was subjected to distillation; it remained clear till about half of it had come over: but then it became rather turbid, and grew more so to the end of the distillation. There remained in the retort apparently four or five grains of brown sediment, that adhered very closely to the bottom and sides of it, nearly half as high as the liquid reached.

1st, This acetous acid, in which JAMES's Powder had been boiled, and afterwards distilled, was found to contain no earth, salt, or metallic matter: nor did the acid itself appear to be altered in its chemical qualities.

2dly, The residuum in the retort had no taste. It did not appear to dissolve by trituration in one ounce of distilled water, nor in lixivium of alkali of tartar, mild or caustic. After boiling this residuum in one and a half ounce of water, part of it seemed to be dissolved; and this solution being filtered was examined with the following re-agents.

(a) Acid of sugar rendered it turbid, and transparency did not ensue on adding a further quantity of this acid.

(b) Muriated barytes produced a considerable turbidness, which was not removed by adding concentrated acetous acid.

(c) Nitrated silver induced a slight turbid appearance.

(d) Mild alkalies induced no visible change.

(e) Prussiated

(e) Prussianated fossil alkali occasioned a very flight opal coloured appearance, and after standing six days a very small quantity of whitish sediment was thrown down, which dissolved on concentrated acetous acid being added, and the liquid turned greenish; but, after standing, a little greenish sediment was deposited, not, however, apparently more than was produced by a mixture of this Prussian alkali, acetous acid, and water.

(f) Phosphorated fossil alkali produced no turbidness or precipitation on standing a week; but on the addition of  $\frac{1}{100}$  gr. of nitrated mercury (which had been made by fully saturating the nitrous acid with mercury) the mixture grew instantly thick, and deposited a copious white sediment.

3dly, The remainder of the residuum, in the retort above mentioned to have been left after distilling to dryness acetous acid in which JAMES'S Powder had been boiled, did not totally dissolve in a large proportion of nitrous acid diluted; but was wholly taken up on adding a little marine acid. A great part of the superabundant acid of this solution being carried off by evaporation, it was examined with the following substances.

(a) Adding a little of it to a large proportion of water, milkiness ensued.

(b) The same appearance took place with a large proportion of lime-water.

(c) A turbid bluish colour was produced on adding Prussian mineral alkali, and on standing a bluish sediment took place.

(d) A polished copper plate was not at all whitened by immersion in this solution; but, on adding to it  $\frac{1}{100}$  of a grain of nitre of mercury, the copper was rendered paler coloured.

The deficiency of twenty grains of powder in these experiments with acetous acid must be ascribed partly to the

solution in this menstruum, and still more to the waste in the paper filter, and to its adhering to the retort when first boiled.

These experiments indicated the same kind of substances as the experiments with water, namely, calcareous earth in a combined state; phosphoric acid; calx of antimony and of iron. It appears also, that JAMES's Powder is either wholly or partially soluble in about 300 times its weight of concentrated acetous acid.

### III. *With nitrous acid.*

The 240 grains of powder remaining after the experiments with acetous acid, p. 327. were digested in the cold twelve hours, in three ounce measures and a half of purified and concentrated nitrous acid, diluted with four ounces of pure water, and then distilled with a gentle heat till there remained about two ounce measures. After standing twenty-four hours, about one ounce and a half of clear liquid, which was very corrosive and acid, was drawn off by means of a syphon. The turbid liquid and close white sediment remaining, being mixed with one ounce of distilled water, were poured upon a filter of paper, and hot distilled water was repeatedly poured upon this filter till it passed through almost tasteless. The first portions of the filtered liquid, in quantity ten ounces, being mixed together, were set to evaporate in a glass pan. As soon as the liquor grew hot, the turbidness disappeared; and as the evaporation went on, first clouds, and afterwards portions of sediment, appeared in a clear liquid. The evaporation being continued nearly to dryness, a white, porous, or cellular cake was left, that weighed 129 grains. The liquid obtained

by distillation from the solution which left this mass was found to be merely diluted nitrous acid.

The residuum left on the filter which had resisted solution in nitrous acid, being well dried, weighed a little more than 142 grains. This residuum was digested, and boiled as before in nitrous acid; and this menstruum, distilled from the residuum, being evaporated to dryness, afforded 6,5 grains of a whitish mass. The residuum left on the filter after this second application of nitrous acid, being well dried, weighed 132 grains.

The mass of 129 grains, left on evaporation of the above solution of JAMES'S Powder in nitrous acid, in a few hours began to deliquesce, especially at the edges. Some of the deliquescent part of the mass was dissolved in one ounce and a half of water, forming an opal-coloured solution, with a white sediment. This opal-coloured solution being filtered was examined.

(a) It rendered lime-water milky; and the milkiness did not disappear on adding concentrated acetic acid, but readily on pouring into the mixture a little acid of nitre.

(b) It turned thick and white, and soon deposited a copious sediment of white matter, with a few drops of nitrous solution of mercury; and became turbid also with nitrated silver.

(c) With muriated barytes it became very turbid, and remained so after adding acetic acid; but grew clear again on adding nitrous acid.

(d) Acid of sugar produced a turbid appearance.

(e) Caustic volatile alkali produced very little precipitation; but a copious one took place with mild alkali of tartar; which precipitated matter, after decanting the clear solution, was nearly all dissolved by acetic acid, and the remainder was readily taken up by the marine as well as by the nitrous acid.

(f) Prussian

(f) Prussian alkali occasioned a light blue colour, but no turbid appearance till the mixture had stood several days, which was then slight.

(g) No change of colour was produced on a copper plate.

A little of the soft and deliquescent part, just mentioned to have taken place at the edges of the solid mass, tasted bitter and sour. It melted under the blow-pipe into a horny kind of globule; but the dry part of this mass could not be fused by this means.

This ounce and a half of solution being consumed in these trials, the same quantity of boiling distilled water was poured on the precipitate or part not dissolved by this quantity of water on the first affusion. After standing and boiling, the precipitate appeared to be but little diminished. The clear liquid did not as before render lime-water turbid; but a precipitate ensued with nitrated mercury, which on comparison was found to be a more delicate test of phosphoric acid than lime-water. This solution also, by this second affusion of water, did not as before grow thick with mild alkali or tartar, nor blue with Prussian alkali. The sediment, undissolved by these two applications of water, did not totally dissolve in a superabundant quantity of nitrous acid; but completely and immediately in a smaller quantity of marine acid; and this solution in marine acid, with a large proportion of water, produced milkiness; with Prussian alkali, it turned of a deep blue colour; it did not whiten copper; saccharine acid and saluted barytes only slightly disturbed its transparency.

By these experiments I found the solution of JAMES's Powder in nitrous acid contained, probably, a pretty considerable proportion of calcareous earth united to both nitrous

and phosphoric acid; a little phosphoric acid in a free state; and a small proportion of calx of antimony and of iron.

Ninety grains of the dry part of the above mass of 129 grains, p. 329. were repeatedly triturated and digested in alcohol till almost nothing was taken up by it. This solution, being filtered, was evaporated to dryness, and afforded 20 grains and a half of a fine white salt, very bitter, which, on exposure to the air, soon became liquid, but very turbid.

The powder that had thus ceased to yield any thing further to alcohol was repeatedly triturated and boiled in pure water, till the liquid passed tasteless through the filter; and the filtered liquors, being evaporated, left eight grains more of a less bitter and less deliquescent substance than that from alcohol. Part only of these eight grains was soluble readily in water; and they appeared to be a mixture of the saline matter dissolved in alcohol, and of the insoluble residuum in that menstruum.

The powder remaining on the filter after these solutions in alcohol and water being dried, weighed nearly 59 grains. It was white and tasteless.

I next examined these products more particularly; and *first* the twenty grains and a half which had been dissolved in alcohol.

(a) With a large proportion of water it produced a rather turbid appearance, and, after standing, a sediment of calcareous earth was deposited.

(b) This last solution (a) being filtered, with mild alkalies grew very thick, and deposited a sediment that was readily taken up by acetic acid.

(c) With caustic volatile alkali its transparency was scarcely disturbed.

(d) With acid of sugar it became thick and white; and

(e) White



(e) White as cream of milk with phosphorated mineral alkali; the sediment from which mixture did not dissolve in a quantity of boiling water that would have dissolved vitriolated lime, nor in vinegar, but was readily taken up by nitrous acid.

(f) The transparency of nitrated and muriated barytes was scarcely disturbed.

(g) It turned infusion of turnsole red.

(h) A little of the deliquescent salt above mentioned, that had been dissolved in alcohol, being made nearly dry, on adding to it a mixture of alcohol and acid of vitriol, vapours of nitrous ether were detached with ebullition

(i) With lime-water it produced a slight sediment.

(k) With Prussian alkali at first a pale green colour, and afterwards a blue colour was produced; but without any precipitation on standing.

(l) This substance, which had been dissolved in alcohol, was infusible under the blow-pipe; and after being heated red-hot on charcoal it was no longer soluble in water. Being further examined, it was found to be merely calcareous earth.

This soluble part then in alcohol appeared to be nothing but nitrated lime, with some traces of calcined iron.

*Secondly,* The 59 grains of powder, not soluble in alcohol, were examined.

(a) A mixture of vitriolic acid and alcohol detached from this powder no nitrous ether, nor any vapour that formed white clouds with volatile alkali.

(b) It did not effervesce, and required above 200 times its weight of concentrated acetous acid to dissolve it.

(c) Under the blow-pipe it emitted no smell or fume, and with great difficulty melted imperfectly, affording an irregular figured, horn-like, opaque mass.

(d) It

(d) It was not diminished or altered in its properties by boiling in lixivium of alkali of tartar.

(e) Nitrous acid formed with it, without effervescence, a very slightly turbid solution; which solution produced the same appearances as those related, p. 330, 331.

(f) 50 grains of this powder were dissolved in nitrous acid, and a great part of the redundant acid being carried off by evaporation, to one half of this solution was added lime-water till it ceased to produce any milkiness, and the mixture tasted of lime-water. After standing excluded from the air, the sediment deposited from a clear liquid was collected, and being dried it weighed 26,3 grains. This precipitate had the properties of phosphorated lime, with that proportion of lime and phosphoric acid which forms a compound scarcely fusible. The liquid in which this precipitate fell seemed to contain a little phosphorated lime, but principally calcareous earth.

(g) To the other half of this solution in nitrous acid (f) was added vitriolic acid, drop by drop, till it no longer disturbed its transparency. After standing, the clear liquid was decanted from the precipitated matter which had taken place, and the precipitate with a small quantity of water was thrown upon a filter. The filtered and decanted liquids mixed together were boiled till the smell of nitrous acid ceased, and there remained about half an ounce of acid liquor, which being filtered to separate the vitriolated lime precipitated during evaporation, fossil alkali was added to perfectly saturate it. During this union there was an effervescence and a separation of more vitriolated lime, which being removed, the saturated liquor, by crystallization, afforded nearly 26 grains of crystals of phosphorated fossil alkali, be-  
fides

sides a little cubic nitre, vitriolated fossil alkali and iron, with some vestiges of calx of antimony, and phosphorated lime.

The precipitate thrown down on adding vitriolic acid, and left upon the filter, weighed when dried 26,1 grains, and was vitriolated lime, with a minute portion of calcined antimony and iron.

The 6,5 grains, p. 330. left on evaporation to dryness of the second solution in nitrous acid, consisted of nearly three grains of calcined antimony, and the rest phosphorated lime, with a little iron.

1. It appears from the above experiments with nitrous acid, that this menstruum, by two affusions, in a large proportion, aided by trituration, digestion, and heat, dissolved  $\frac{1}{2} \frac{0}{4} \frac{8}{0}$  of JAMES's Powder that had been exposed to the action of water and acetous acid; but from the smallness of the quantity contained in the nitrous acid the second time it was applied, and from its being principally calcined antimony, not more than two of the six grains afforded by this solution, perhaps, should be considered to be *dissolved*, for the rest may be supposed to be merely *suspended*.

The first solution also in this menstruum was not filtered, and the acid was considerably redundant, and there was found in it several grains of calcined antimony. The real quantity *dissolved* might therefore probably be eight grains less than the above 108 stated. According to this mode of calculation, the proportion of the soluble part of JAMES's Powder in nitrous acid is  $\frac{1}{2} \frac{0}{4} \frac{0}{0}$ , or about  $\frac{4}{100}$ .

2. The whole of this soluble part, except a little calx of antimony, is, decisively, phosphoric acid and calcareous earth :  
which

which two substances may reasonably be supposed, from these experiments, to have been united together, and to have been in the state of phosphorated lime in this powder. Consequently, the proportion of this phosphorated lime, considered as the soluble part of JAMES'S powder in these experiments with nitrous acid, appears to be *40 per cent.* making a deduction of *1 per cent.* for the antimonial calx contained in the nitrous acid in the above experiments. It is however already obvious to suspect, that the powder which resisted solution in this menstruum may contain more phosphorated lime; and this consideration prevents me assigning at present the above *40 per cent.* as the whole quantity of it in JAMES'S Powder. It cannot however, I think, be a smaller proportion.

I do not reckon the calx of iron in these calculations, because it is in too small a quantity, and is apparently only to be looked upon as an accidental extraneous substance. I suppose too, that the water and acetous acid applied to the JAMES'S Powder used in these experiments, carried off a proportion of its ingredients equal to that in the remaining powder.

#### IV. *With marine acid.*

The 132 grains of heavy, white, tasteless powder, the residuum after boiling 240 grains of JAMES'S Powder in nitrous acid, till it had dissolved that part for which it has any considerable affinity, p. 330. were digested for twenty-four hours in eight ounce-measures of marine acid, the specific gravity of which was 1,170, and diluted with half its bulk of distilled water. This mixture was distilled in a

gentle heat till there remained about two ounce-measures of a very turbid liquor. After standing in a jar two days, it deposited a close white sediment, obviously much less in bulk than the powder added to this menstruum; and nearly one ounce measure and three quarters of clear yellow liquid were drawn off, by means of a siphon, which was marked N° 1.

The distilled liquid, which was merely diluted marine acid, was poured back on the sediment and remaining liquid; and after digestion twenty-four hours, this mixture was distilled as before, till there remained about one and a half ounce-measure; but after standing in a jar several days, the quantity of sediment deposited was apparently as great as before the second application of this menstruum. The clear liquor was drawn off as before, and marked N° 2. The distilled liquor being found to be merely diluted marine acid, was poured on the remaining liquid and sediment a third time; and, after digestion, the distillation was repeated as before. The remaining liquid having stood upon the sediment some time, one ounce-measure of clear liquor was drawn off, and marked N° 3. The sediment did not appear diminished by this third distillation; but, as the decanted liquid, N° 3. was found to contain a small quantity of some substance dissolved or suspended in it, the marine acid distilled in this experiment was poured a fourth time on this residuum, and after digestion boiled. Having stood several days, the clear liquor was decanted, and marked N° 4. To the residuum, after these four affusions of marine acid, one ounce of boiling distilled water was added, and this mixture was poured on a filter. The powder upon the filter being well dried, was found to weigh 60,1 grains.

In the ounce of boiling water that had been filtered from this residuum, I could find nothing but some minute particles of that substance and vestiges of iron. Two or three drops of the liquor, N° 1. added to three ounces of distilled water, produced a pretty considerable milkiness; and, on standing, a close white sediment was deposited. Two or three drops of the liquor, N° 2. produced less milkiness and sediment in the quantity of water just mentioned than N° 1. N° 3. scarcely diminished the transparency of distilled water; and N° 4. did not affect it all. The liquids, N° 3. and 4. were distilled till there remained about two drachm-measures of clear brown liquid, with a cloudy sediment. N° 3. being poured into two ounces of distilled water scarcely made it milky; and N° 4. did not diminish the transparency of water at all. On evaporating to dryness these mixtures of the liquors, N° 3. and 4. with distilled water; that with N° 3. left a yellowish dry sediment which weighed 3,1 grains; and that of N° 4. left 2,2 grains of sediment. Nothing but calx of antimony could be discovered in these sediments. They were reducible readily with tartar; scarcely fusible in the spoon; but with phosphoric acid easily melted into an opaque yellowish globule.

The liquor, marked N° 1. being poured into twenty-four ounces of distilled water produced a very milky appearance; and the same appearance, but in a much less degree, ensued on pouring the liquor N° 2. into this quantity of pure water. After standing several days, a white sediment being deposited from a clear watery fluid, the clear liquid was drawn off by means of a siphon, and the sediments being dried, that of N° 1. weighed 51 grains; that of N° 2. weighed 6,15 grains; and both were found to be purely Algaroth powder. The water, *viz.* 4<sup>8</sup> ounces, in which these precipitates fell, being  
 evaporated

evaporated to dryness, left a little more than four grains of an infusible kind of sediment, which was calx of antimony, like that of N<sup>o</sup> 3. and 4. with a minute portion of Algaroth powder and iron.

The 60,1 grains, p. 337. which resisted solution in marine acid, were a lighter powder than JAMES's Powder itself, rather gritty, quite tasteless. This powder was digested in one-ounce measure of concentrated nitrous acid mixed with two of acid of salt for a week; and then this mixture was diluted with four ounces of distilled water, and gently boiled till it was reduced to about two ounce-measures. On cooling and standing several days, a sediment of the same kind apparently, and in the same quantity as before evaporation, was deposited, and, after standing, a clear yellow liquid was drawn off. The sediment, well dried, weighed 55 grains. The decanted liquor being reduced by evaporation from one and a half ounce to about two drachm-measures, a sediment, while hot, appeared, which was calx of antimony, with a minute portion of earthy matter that had some properties of phosphorated lime and calx of iron. A little of the clear liquor on this sediment being dropped into water produced no milkiness, nor sediment, after standing; but the whole of this clear liquor, with sediment, being evaporated to dryness, afforded four grains of the same kind of infusible calx of antimony, mentioned to have been obtained from the acid liquors, N<sup>o</sup> 3. and 4. above mentioned.

It appears then, that by repeatedly digesting and boiling in marine acid, and in *aqua regia*, that part of JAMES's Powder which resisted solution in nitrous acid, which was  $\frac{1}{2} \frac{3}{4} \frac{2}{8}$ , p. 335. 77 grains were carried off by these menstrua; but considering the small proportion contained in these acids after the two first

affusions, which afforded 57,15 grains, p. 338., and supposing the calx to be neither increased nor diminished in weight by the acids, the real quantity of soluble and fusible calx of antimony may be stated to be that of Algaroth powder; for the other kind of antimonial calx obtained by subsequent affusions was probably only *suspended*. Consequently 240 grains of JAMES's Powder afforded, by the above experiments with marine acid, 57,15 grains of Algaroth powder, and 19,85 grains of a less soluble and more difficultly fusible calx of antimony, with a small proportion of phosphorated lime. The residuum, amounting to 55 grains, was of course next examined.

*Experiments upon the part of JAMES's Powder which was insoluble in the above menstrua.*

(a) A few grains of this insoluble substance could neither be melted nor carried off in vapour by means of the flame of a candle applied to it upon charcoal, and also in the spoon, with the blow-pipe; but,

(b) Mixed with an equal weight of tartar, it melted on charcoal; and, while in fusion, small, apparently, metallic grains were distinctly perceived; and on cooling they could be seen, even without a lens, adhering to an irregularly figured, opaque, whitish mass. Sometimes slight explosions were heard while the flame was applied. The metallic grains appeared more distinctly when this powder was mixed with one-third of its weight of powdered calcined bone, than in JAMES's Powder.

(c) Ten grains of this powder were melted as above mentioned, by repeatedly applying flame with the blow-pipe to  
two.



two or three grains of it at a time mixed with tartar. The opaque whitish masses, with metallic grains in them, thus obtained, being pulverized, were digested and gently boiled in diluted nitrous acid. The filtered solution afforded nitre, and nitrous acid in a free state, the greatest part of which superabundant acid being carried off, the lixivium did not whiten copper, or throw down any calx but iron with Prussian alkali. The residuum that resisted solution in nitrous acid was digested and gently boiled in *aqua regia*. On standing it was decanted, and this decanted liquid being heated, to carry off superabundant acid and water, it afforded on mixture with water 1,2 grain of Algaroth powder, and no metallic matter could be detected in the water excepting a little iron. A small part only being dissolved by the *aqua regia*, the residuum was exposed to the flame of a candle with tartar as before; and, by the aid of a lens, I could just perceive two or three metallic grains in the fused mass. To this mass the *aqua regia* was again applied, and 0,15 grain of Algaroth powder was obtained, and no other metallic calx was found but iron. A third affusion of *aqua regia* indicated an exceedingly minute portion of Algaroth powder; but I could afterwards perceive no more metallic grains in the residuum exposed to flame with tartar, nor obtain more Algaroth powder from the solution of the fused mass in *aqua regia*. The residuum that resisted solution melted readily with a little phosphoric acid into an opaque, somewhat yellowish, white globule, not unlike calcined bone fused with phosphoric acid, and a minute portion of flowers of antimony. The quantity, however, of this residuum was so small, that I despaired of determining its nature further by more experiments.

Having

Having found that this insoluble powder would not melt with sulphur when heated *red-hot*, I made the following experiment.

(*d*) Twenty grains of it being mixed with three times its weight of sulphur, were put into one of Mr. WEDGWOOD's crucibles that would contain one ounce-measure, which, with a cover well luted on, was put into a three-inch English crucible, and calcined bone in powder filled the space between the two crucibles. After exposing this charge to a red heat half an hour, and in a white heat ten minutes, the crucibles were cooled; and being opened, the pyrometer piece of WEDGWOOD in the bone ashes was found to indicate  $65^{\circ}$ , and the mixture in the inner crucible had apparently been melted into a resin-like mass that adhered firmly to the sides of the vessel. Twenty-eight grains were scraped off, which, after digestion and boiling in marine acid, afforded six grains of Algaroth powder. A great deal of hepatic air was discharged during this solution, and very little sulphur was left on the filter with the part not dissolved by the marine acid. This undissolved part, which weighed six grains, was blackish, tasteless, not heavy. It was infusible with the blow-pipe, both alone, and mixed with sulphur and tartar; but with phosphoric acid it melted into a blackish scoria-like mass. I could only conjecture, that this last part was antimonial calx, so far vitrified with phosphorated lime as to be neither soluble nor reducible or fusible, except with phosphoric acid.

(*e*) By a similar experiment, but with alkali of tartar twenty grains, sulphur thirty grains, and ten grains of this insoluble part of JAMES's Powder, a fused mass was obtained that partially dissolved in water, and afforded kermes mineral on pouring an acid into this solution; but a great part was  
insoluble

insoluble in water and acids, and seemed to be of the same nature as the six grains of residuum just mentioned (*d*).

I could only conclude from these experiments on this insoluble matter, that it contained calx of antimony; but as to the proportion of it, and the other substance with which it is joined, I conjecture that it may be about half the quantity of the insoluble powder; and that the other half is antimonial calx and phosphorated lime, so highly calcined and vitrified together as to resist solution in acid menstrua, decomposition by charcoal, and fusion with fixed alkalies, but not by phosphoric acid.

I should not have been satisfied with here terminating this analysis without enquiring further into the nature of this insoluble matter; but I discontinued this analytic investigation in order to derive light from the synthetic experiments which will be related hereafter.

These last experiments seem to shew, that the proportion of antimonial calx is not so great as might have been assigned from the experiments with nitrous acid, marine acid, and *aqua regia*.

The substances and proportions of them, obtained from 240 grains of JAMES's Powder, by the above experiments, are as follow :

	Grains.
Phosphorated lime, with a little antimonial calx,	100,
Algaroth powder, . . . . .	57,15
Insoluble antimonial calx, with a little phosphorated	
lime, . . . . .	19,85
The same insoluble calx, with, probably, a little phos-	
phorated lime, . . . . .	55,
Waste, . . . . .	8,
	<hr/>
	240,0

As

As it may be objected, that conclusions drawn concerning the nature of calces might be erroneous if nitrous acid had been applied previously to substances containing them, I made the following experiment.

*Experiment with marine acid applied to JAMES'S Powder, which had not been exposed to the action of nitrous acid, or any other menstruum.*

50 grains of JAMES'S powder were digested, and gently boiled in two ounce-measures and a half of concentrated marine acid diluted with one ounce of distilled water till there remained only about one ounce-measure. A great part of the powder appeared to be evidently dissolved. On cooling, crystals of muriated antimony were formed upon a white sediment. The clear liquid with the crystals being decanted, the sediment was boiled twice, as before, with marine acid; but the second affusion of this menstruum brought off but eight grains of this powder, and the third only four grains. The remaining sediment, being well dried, weighed 14 grains. Now it has been shewn already, that the nitrous and marine acids, successively applied, dissolved  $\frac{1\ 7\ 7}{2\ 4\ 0}$ , or all but about 60 grains; and in the present experiment, the marine acid dissolved  $\frac{3\ 6}{3\ 0}$ , which is in the proportion of  $\frac{1\ 8\ 0}{2\ 3\ 0}$ , or nearly  $\frac{1\ 7\ 3}{2\ 4\ 0}$ ; so that, on account of the trifling difference in these proportions, it may, perhaps, be fairly concluded, that the properties of the calx in JAMES'S Powder are not altered by nitrous acid to affect its solubility in marine acid. And further, this insoluble powder in the present experiment was found to have the same properties as that in the former experiments.

To

To know whether JAMES's Powder contained any substance that could be decomposed by mild fixed alkalies, the following experiments were made.

These experiments with fixed alkali seemed to be especially necessary, because phosphoric acid, lime, and antimonial calx, are ingredients in JAMES's Powder; and it was obvious to suspect, that this acid might be united with calx of antimony as well as with lime; which phosphorated antimony would be decomposed by alkalies, and yield phosphorated alkali.

*Experiments with fixed alkalies.*

100 grains of JAMES's Powder were boiled in six ounces of water, with 50 grains of mild alkali of tartar, for three hours, and then the remaining liquid was filtered, and evaporated to dryness; but the matter left after evaporation was nothing but the alkali used in the experiment, with a little of the powder itself.

The result was the same on making the experiment with crystallized mineral alkali instead of alkali of tartar.

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*Synthetic Experiments.*

ALTHOUGH the inability to prepare JAMES's Powder would not prove the above conclusions, with respect to its composition, to be erroneous; the being able to compose a substance

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possessing all the same properties as JAMES's Powder, by uniting or mixing together the substances shewn by the above analysis to enter into its composition, would afford all the proof and demonstration which can be had in the science of chemistry.

The above analysis shewed no essential ingredients of JAMES's Powder but antimonial calces, phosphoric acid, and calcareous earth, which two last substances appeared to be united together; but it would have been vain and unnecessary labour to have attempted to make this powder by mixtures of any of the commonly known calces of antimony and phosphorated lime; because none of them, from their well known qualities, could form a powder of the same colour and specific gravity as JAMES's Powder, and like it partially soluble in acids. From the above experiments, however, the probability was evident, that this substance might be made by calcining together antimony and bone-ashes; which operation produces a powder called LILE's and SCHAWANBERG's fever-powder; a preparation described by SCHRODER and other chemists 150 years ago. The receipts for this preparation differed in the proportion of the antimony to the bone ashes, and in the state of the bone; some directing bone shavings to be previously boiled in water; others ordered them to be burnt to ashes before calcining them with antimony; and in other prescriptions the bone shavings were directed to be burnt with the antimony. According to the receipt in the possession of Mr. BROMFIELD, by which this powder was prepared forty-five years ago, and before any medicine was known by the name of JAMES's Powder, two pounds of hart's horn shavings must be boiled to dissolve all the mucilage, and then, being dried, be calcined with one pound of crude antimony, till the smell of sulphur

fulphur ceases, and a light grey powder is produced. The same prescription was given to Mr. WILLIS, above forty years ago, by Dr. JOHN EATON, of the College of Physicians, with the material addition, however, of ordering the calcined mixture to be exposed to a great heat in a close vessel to render it *white*. Mr. TURNER made this powder above thirty years ago by calcining together equal weights of burnt hart's horn and antimony in an open vessel, till all the fulphur was driven off, and the mixture was of a light grey colour. He likewise was acquainted with the fact, that by a sufficient degree of fire in a close vessel this cineritious powder turned *white* \*. Mr. TURNER also prepared this powder with a pound and a half of hart's horn shavings and a pound of antimony, as well as with smaller proportions of bone. SCHRODER prescribes equal weights of antimony and calcined hart's horn; and POTERIUS and MICHAELIS, as quoted by FREDERIC HOFFMAN, merely order the calcination of these two substances together (assigning no proportion), in a reverberatory fire for several days. In the London *Pharmacopœia* of 1788, this powder is called *Pulvis antimonialis*; and it is directed to be prepared by calcining together equal weights of hart's horn shavings and antimony.

Powders made from various proportions of antimony and bone-ashes, after solution in nitrous acid, left a residuum of antimonial calx much less or greater in quantity than JAMES's Powder did by the same menstruum, except two of Mr. TUR-

\* It is probable, that this powder was made for several years with merely the heat necessary to carry off the fulphur and calcine the bone, in an open vessel over a charcoal fire in a common grate, and consequently it was of a light clay or ash colour. In this manner, Mr. BROMFIELD told me, he prepared SCHAWANBERG's Powder 46 or 47 years ago. Its property of turning *white* in a greater degree of fire appears to have been a subsequent discovery.

NER's proportions, viz. two parts of antimony and one of calcined bone, and equal weights of bone shavings and antimony. The quantity of this calx was, however, greater in the powder from the former of these two last proportions than the latter of them; which latter corresponded sometimes exactly, and always nearly, with the weight of the calx from a given weight of JAMES's Powder. This calx afforded also the same proportion of Algaroth powder as the calx in JAMES's Powder; and the insoluble part of the calx afforded metallic grains like those from the insoluble part of the calx in that powder.

I found then an exact correspondence between what I consider to be the essential and peculiar properties of JAMES's Powder, and the properties of a powder made by uniting or mixing together the ingredients of JAMES's Powder found by analysis. But, in order to shew the identity or difference of the qualities of these two substances, I made comparative observations on them, and repeated the above analytic experiments on JAMES's Powder with the preparation made by calcining together equal weights of bone shavings and antimony, in an open vessel, to carry off the sulphur, and then in close vessels applying such a degree of fire as to render them *white*, that is, on the same preparation as the *Pulvis antimonialis* of the London *Pharmacopæia*.

First, I compared, more particularly, the sensible qualities of several different specimens of JAMES's Powder with various parcels of the *Pulvis antimonialis* made by different chemists. All of these would be called white powders, but not two of them were so in the same degree. Most of the papers of the *Pulvis antimonialis* were whiter than those of JAMES's Powder; but others were of a very light stone colour, and some had a shade of yellow, so as to resemble very exactly JAMES's Powder;

but



but all the parcels of JAMES's Powder had either a shade of yellow or of stone colour, and none were perfectly white, or so white as some specimens of the *Pulvis antimonialis*. Some of the parcels of JAMES's Powder and of the *Pulvis antimonialis* tasted brassy; and other specimens of both powders had no taste. All of these powders were gritty. Most of the parcels of the *Pulvis antimonialis* were a little specifically heavier than those of JAMES's Powder. The specific gravity of both powders was increased by exposing them to such a degree of fire as brought them into almost a semi-vitrified state; and, on the contrary, the specific gravity of the *Pulvis antimonialis* was less than it is in its usual state, when made in such a degree of fire that the mixture preserves the powdery form.

The experiments with water on the *Pulvis antimonialis* produced the same kind of appearances, but more slightly than those with JAMES's Powder; for the hot solution of the former grew less milky on cooling than that of the latter, and on evaporation to dryness less sediment was found of the solution of *Pulvis antimonialis* than after that of JAMES's Powder\*.

The experiments with acetous acid on the *Pulvis antimonialis* shewed, that this menstruum dissolved sometimes a greater, and sometimes a smaller proportion of it than of JAMES's powder; and the dissolved matter was found to be antimonial calx, phosphorated lime, and calx of iron, and no other substance.

It has been already said, that the proportion of soluble matter in nitrous acid was the same, or nearly so, of the *Pulvis antimonialis* as that of JAMES's Powder; and this dissolved matter was phosphoric acid, calcareous earth, with a little antimonial calx, and a minute portion of calx of iron, as exactly

\* The reason for this difference is assigned in another place.

as could be expected from the nature of the substances and the experiments, in the same proportion as those in JAMES's Powder.

The Algaroth powder, obtained by means of solution of the *Pulvis antimonialis* in marine acid, was in the same proportion as nearly as could reasonably be expected from the nature of the experiments as that obtained from JAMES's Powder. And the part that resisted solution in this menstruum was partially reducible to a metallic form, and had otherwise the same properties, as far as discovered, as the insoluble part of JAMES's Powder.

Having now formed a powder possessed of properties similar in kind to every one of those ascertained in JAMES's Powder, with scarcely any difference in the *degree* of them, if it be thought that among these properties are those which are essential and peculiar ones of JAMES's Powder, the conclusion that these two are the same kind of things must be admitted to be just. The nature of one of the ingredients of JAMES's Powder, *viz.* the irreducible part of the insoluble matter, p. 342. is not fully elucidated by the synthetic experiments; but in so far as they shew, that this part equally exists in the powder formed by calcining together antimony and bone, which is concluded to be JAMES's Powder, the objection against the conclusion with respect to the identity of the two substances, on the ground of this inconsiderable part of JAMES's Powder not being well understood, must be of little weight.

Several reasons, more interesting to myself than to the Society, induced me to authenticate by additional testimonies those analytic experiments, which may be considered to be more decisive than the rest for establishing the identity of JAMES's Powder, and a powder formed by calcining together antimony and bone-ashes. I therefore requested Mr. CAVALLO and Mr. TURNER to be

present when I made those experiments on the *Pulvis antimonialis*, prepared by Mr. GRIFFIN, of Apothecaries' Hall, and JAMES's Powder. Having, in the presence of these two Gentlemen\*, broken the seal of a phial of JAMES's Powder, bought of F. NEWBERRY, and taken out of it the quantity required for the experiments, the bottle was again sealed by Mr. CAVALLO with his seal, as well as the phial from which was taken the *Pulvis antimonialis*. Should any experiments be published, which establish different conclusions from those contained in this Paper, with respect to the identity of these two powders, I shall be happy to endeavour to ascertain the truth by experiments, on the remaining parcels of the two powders, in the presence of competent judges.

I shall next relate the experiments made with the view of confirming or invalidating the conclusions drawn from the above analysis, with respect to the ingredients and proportions of them in JAMES's Powder; and by which I especially endeavoured to make such antimonial calces as this substance contains, by processes different from those above related.

EXP. I. (a) Hart's horn shavings, of six different parcels, well dried, separately calcined in the same manner, and apparently to the same degree as when calcined with antimony to make LILE's Powder, afforded a light brown coarse powder, with a few thin light black pieces, and lost from 43 to 48 *per cent.* of their weight. The mean loss of weight, of course, was  $45\frac{1}{2}$  *per cent.*

(b) This calcined bone (a), being pulverized, was exposed to a greater degree of fire, in close vessels, than that necessary to render the calcined mixture of antimony and bone-ashes *white*. The loss of weight by this second calcination or exposure to fire was from two to three *per cent.*; and the ashes

\* DR. CLARKE also was present at the beginning of these experiments,

were as white as snow. The total mean loss of weight, by these two calcinations, was then  $\frac{4.8}{100}$ .

EXP. II. 2000 grains of coarsely powdered antimony were calcined in an earthen dish, as in making LILE'S Powder, by constantly raking them about for above three hours. During a great part of this time the vessel was red hot at the bottom; and for the last hour the sulphureous fumes had entirely ceased. The calx thus produced was of a pale bluish colour; it melted, in a low degree of heat, into an opaque, scoria-like brittle mass; it yielded no hepatic air with marine acid; it weighed 1409 grains, or the antimony lost nearly  $29\frac{1}{2}$  *per cent.* The pyrometer in the vessel with the antimony during its calcination, was contracted to the 6th degree of WEDGWOOD'S scale.

The sum therefore of the loss of antimony and bone by calcination in this manner, separately, was  $37\frac{1}{2}$  *per cent.* These two substances were in the next place calcined together in the same manner in an open vessel, as above mentioned.

EXP. III. 2000 grains of antimony from the same parcel as that in the last experiment, and an equal weight of hart's horn shavings taken from the same parcel as those were in Exp. I. were calcined together in the same manner that these substances had been separately. During the first quarter of an hour, the mixture smoked, was black, smelled strongly of sulphur, and felt soft. For half an hour more, the smell of sulphur continued, the mixture turned brown, and the bone was reduced to ashes. At the end of this time, not only the bottom of the vessel might be kept red hot without any signs of fusion; but the smell of sulphur, though weakly, continued for half an hour more in a heat to keep a great part of the mixture red hot. At this time the sulphureous smell rather suddenly disappeared, and could not be perceived, though a  
little

little of the mixture was made quite red hot for a quarter of an hour further; during which no fume was seen, or smell perceived. After cooling, a light grey or cineritious heavy powder was left; on examining which, argentine *spicula* were seen in the larger grains of this calcined substance. It weighed 2200 grains, therefore the loss of weight was 45 *per cent.* The WEDGWOOD pyrometer pieces indicated 8°. In other similar experiments the loss, by calcination, was from 37 to 41 *per cent.*; therefore the mean proportion lost in these experiments must be stated at 41 *per cent.*

It appears, that the calcination of antimony with bone-ashes is much more speedy than when by itself, but the degree of fire was a little greater in the last experiment than in that with antimony alone. Considering the nature of these experiments, perhaps, it may be more reasonable to impute the 3½ *per cent.* greater loss in this last experiment than the sum of the loss in Exp. 1. and 2. to the greater insensible sublimation of the calx from more fire in one case than in the other, than to refer it to the larger quantity of air combined with the metal in the former of these two last experiments.

EXP. IV. The above light clay or ash-coloured powder, obtained in the last experiment by calcining together antimony and bone, being exposed to various degrees of fire from 20° to 165° of WEDGWOOD's pyrometer, in close crucibles, was not at all increased in weight, but generally lost about 5 *per cent.* when a pretty large quantity, as a pound, was in the vessel. A part of this loss must be referred to the adhesion or vitrification of the charge with the sides of the crucible, and part to the deficiency of the bone itself, as above shewn, by further exposure to fire. I am sensible, that in experiments of this nature all calculation must necessarily, to a certain degree,

be vague; yet it may be of some application to observe, that the proportion of antimonial calx, estimated to be contained in LILE's Powder or *Pulvis antimonialis*, and JAMES's Powder, p. 343. from the analysis of them, does not differ more considerably from the proportion of this calx than may, perhaps, be reasonably expected on calculation from these four last experiments to exist in them: for  $70\frac{1}{2}$  parts of antimonial calx, p. 352. to  $54\frac{1}{2}$  parts of bone-ashes, p. 351. is as about 56,4 parts of this calx. to 43,6 parts of calcined bone; and, on analysis, JAMES's Powder afforded  $\frac{57}{100}$  of antimonial calx, and  $\frac{43}{100}$  of phosphorated lime, or nearly so, p. 343. allowing for the waste.

EXP. v. This experiment shews the degree of fire necessary to render the antimony calcined with bone of a white colour; and that this whiteness does not depend on the air, but on the fire.

(a) 1500 grains of the calcined mixture of antimony and bone, Exp. 3. were kept red hot in a close vessel for half an hour. On cooling, I found the powder changed from a cineritious or clay colour to a whitish colour with a shade of yellow. The sides of the crucible were not glazed. The pyrometer in the middle of the powder had contracted to  $40^{\circ}$ . This powder was much inferior in whiteness to JAMES's Powder, being much yellower.

(b) Another parcel of the same powder, Exp. 3. was exposed in the same manner, but to a greater degree of fire, in which the crucible was almost white hot for half an hour. After cooling, the powder was found changed to a loosely cohering, snow-white, heavy mass, and the sides of the crucible were covered with a yellow glaze. This mass, which

was

was easily detached from the vessel, was found covered with a yellow vitreous coat over the whole surface of it that had been in contact with the crucible. In the white solid, on breaking it, many argentine *spicula* were seen. The pyrometer used in all these experiments indicated  $71^{\circ}$ .

(c) 1500 grains of the same parcel, Exp. 3. were exposed in an open crucible to the fire of a melting furnace; no fumes arose till the crucible began to be almost white hot. After inverting another crucible, with a small hole in its bottom, the fumes continued to ascend at times through the aperture for a quarter of an hour. The crucible was then taken out of the fire, and on cooling a *whitish powder* was found, but no glazing, and the pyrometer indicated  $28^{\circ}$ . On again exposing this crucible with one inverted over it in the melting furnace, but to a greater degree of fire, still more fumes arose; but, on cooling, the charge was still in the state of a powder, though *whiter* than before; and the inside of the inverted crucible was covered with silvery particles, and the hole of it was surrounded with argentine *spicula*, in a stellated form. The pyrometer indicated  $39^{\circ}$ . On reducing a little of this powder to a greater degree of fineness, it was as white as JAMES's Powder, with a yellowish cast like it, but inferior in whiteness to a specimen of *Pulvis antimonialis*. This crucible, containing its charge, with a cover closely luted on it, was put again into the fire, which was raised much higher than before; and, after being exposed in it twenty minutes, the powder in the crucible became a loosely cohering solid, as *white as snow*, with a vitreous yellow coat, as before observed; the inside of the crucible was glazed and covered with *spicula*. The pyrometer-piece in the middle of the powder was also covered with a yellow coat, but not

glazed, and it indicated  $81^{\circ}$ . This loosely cohering solid being pulverized afforded a *whiter* powder than JAMES'S Powder.

(d) The crucible, with its charge (b), having a cover well luted on it, was again put into the furnace, and the fire raised to almost as great a degree as I was able. This intense heat was kept up above an hour. After cooling, a white hard solid mass was found within the crucible. On breaking the vessel, to detach from it the charge, this solid mass was found as hard as marble, and to have received its figure from the crucible. Its surface was covered with a yellow vitreous coat, and the whole inside of the vessel had a beautiful gold-coloured glaze with many argentine *spicula*. The pyrometer piece in the middle of the charge was also covered with a fine yellow glaze, and indicated  $166^{\circ}$ . This solid, hard mass weighed only 21 grains less than before the experiment, though the whole inside of the crucible was glazed, and had shining *spicula* upon it. A piece of this hard mass being pulverized, it afforded a whiter powder than JAMES'S powder is in general.

EXP. VI. 2000 grains of coarsely powdered antimony, mixed with 1105 grains of calcined hart's horn in powder, were calcined first in an open vessel, and then exposed to a great degree of fire in a close vessel, as in the above experiments with bone-shavings, Exp. 3. and 4. The calcination of this mixture in the open vessel afforded 2550 \* grains, of a less whitish and rather yellowish powder, instead of a light ash-colour, as with bone shavings, Exp. 3. p. 353.; and by the second, and even

\* In another experiment of this kind 2400 grains of antimony and 1500 grains of calcined bone afforded 3450 grains of yellowish light-brown powder. In a third trial, 600 grains of antimony and 400 grains of calcined bone gave 850 grains of yellowish brown powder.

repeated



repeated exposure to fire, it never could be made quite so white, but seemed more inclined to melt than the powder prepared with unburnt bone. In other respects the effects of fire were apparently the same, or nearly so, as in the experiments with bone shavings, Exp. 3, 4. ; for though the loss of weight in this experiment, reckoning that of the antimony at  $29\frac{1}{2}$  per cent., and that of the bone ashes at  $2\frac{1}{2}$  per cent. should have left 2483 only, instead of 2550 ; yet, in other similar experiments, the product corresponded nearer to this calculation, and the loss was sometimes less both of the antimony and bone calcined separately. Some of the persons who prepare the *Pulvis antimonialis* say, that the whitest colour is obtained by first boiling the bone shavings to dissolve their mucilage, and then calcining them with antimony as above shewn. Mr. LILE's receipt directs previous decoction of the hart's horn.

It will not be difficult, from these experiments, to give a probable reason for the JAMES's Powder being generally of a yellowish cast, and for different parcels of it, as well as of the *Pulvis antimonialis*, being generally of different degrees of whiteness and shades of yellow. The colour of this preparation is, however, a very delicate one. I once directed a person to calcine together antimony and bone shavings, in the usual manner, to that state in which the white powder may be produced by a due degree of fire ; but, instead of a snow-white mass, I could not by any degree of fire obtain any colour but a dirty whitish or light stone colour ; though repeated calcinations were employed. The reason of the failure was, that the earthen dish had been broken during the calcination, and a few very small pieces of it had scaled off, and being mixed with the powder occasioned this disappointment with

with respect to colour. The same disappointment has been also occasioned by using a rusty iron rod in calcining the mixture.

The bone-ashes procured from the sal ammoniac and spirit of hart's horn manufactories, frequently failed in producing a white powder; and so did sometimes the bone-ashes, called prepared hart's horn, sold by the druggists. Even after a fine white coloured mass had been made, if it was pulverized in an iron mortar that had extremely little calx upon its surface, or dirt, the powder was not white.

The yellow coat and glaze on the sides of the crucible and surface of the calcined mixture of bone and antimony, in these experiments, is to be ascribed rather to the fusion of the clay of the crucible with the antimonial calx, than to the greater degree of fire in the part of the crucible in which it takes place; or than to the calx of iron and siliceous earth of the vessel: because the same yellow coat and glazing are produced on the WEDGWOOD pyrometer pieces, which are placed in the middle of the charge, and where the degree of heat cannot be so great as nearer the side of the crucible, and yet a snow-white mass is produced between these clay pieces and the sides of the crucible. This effect of clay, in forming a yellow coat and glaze, is shewn by the observation of what happens when the calcined mixture is put into a WEDGWOOD's crucible, which is made of much purer clay than other vessels of this kind, and when it is set in a larger Hessian crucible with the space betwixt the two vessels filled with the same calcined mixture. After exposure to a sufficient degree of fire, *viz.* about  $120^{\circ}$  of WEDGWOOD's scale, the inside and outside of the inner crucible will be covered with a yellow vitreous coat and glaze as well as the inside of the outer crucible in contact with the charge, while the rest of the matter within these vessels is of  
a snowy

a snowy whiteness. This yellow coat is one reason for the powder being of a shade of yellow in some specimens.

Supposing the fusibility of the antimonial calces to be diminished the more they are calcined; the following experiment shews, that the antimonial calx in JAMES's Powder is more calcined than that in Exp. 2.

EXP. VII.  $70\frac{1}{2}$  grains of calcined antimony, as prepared in Exp. 2. triturated with  $53\frac{1}{2}$  grains of calcined bone, formed a powder of a bluish cast, which being exposed in a close crucible, for half an hour, in a melting furnace, the degree of fire in which was  $120^{\circ}$  of WEDGWOOD's scale, it was found melted into a vitreous, pale bluish mass; and the inside of the crucible was glazed yellow, with red streaks, and had argentine *spicula* adhering to it.

EXP. VIII. 800 grains of the calcined antimony of Exp. 2. were calcined for eight hours in a dish, as in making LILE's Powder, by stirring it constantly, and keeping the bottom of the vessel red hot during the whole time; the two last hours also the whole of the powder was kept red hot. On cooling, this calx was an impalpable light-brown powder.

(a) 100 grains of this calx, triturated with an equal quantity of calcined hart's horn, formed a powder very unlike JAMES's Powder, for it was of a light-brown colour. On exposing it to about  $120^{\circ}$  of fire it melted into a yellow opaque mass.

(b) The remaining 700 grains of the calcined antimony of this experiment were exposed to fire and air as before for eight hours longer, and kept red hot a great part of the time; but the calx became very little lighter coloured than before.

(c) 100 grains of this calx last mentioned (b), triturated with as much calcined hart's horn, being exposed to the degree of fire usually applied in making the *Pulvis Antimonialis*, in a

close vessel, the mixture melted partially into a greyish mass.

(d) 150 grains of the calcined antimony (*b*) of this experiment were mixed with an equal weight of calcined hart's horn. This mixture was raked about in an earthen dish for an hour, during a great part of which time it was red hot. On cooling, the powder was evidently lighter coloured than before this calcination. It was then exposed in a close crucible to a white heat for half an hour; and, after cooling, a loosely cohering white solid, with a vitreous yellow coat, was found, little inferior in whiteness, and otherwise resembling JAMES'S Powder.

(e) 300 grains of the calcined antimony (*b*) of this experiment were raked about in an earthen dish for an hour, a great part of which time they were kept red hot. On cooling, the calx was found of the same colour as before; and after exposing it in a close crucible in the melting furnace to almost a white heat for half an hour, it was observed to have been melted into a yellowish mass.

It seems at least very probable, from this experiment, that no degree or duration of fire, applied in open or close vessels to antimony *alone*, can produce a calx of the same kind as that in JAMES'S Powder: nor, perhaps, can such a powder be composed by fire applied, in close vessels, to calx of antimony mixed with calcined bone; but if antimony duly calcined be mixed with calcined bone, and exposed to air, in a due degree of fire, for a sufficient length of time, and then a still greater degree of fire be applied to it in close vessels, such a compound may be formed as JAMES'S Powder. This experiment also proves, that the sulphur in antimony is no ways necessary to the formation of this compound.

The manner in which air and fire act upon the antimonial calx and phosphorated lime, I shall venture to conjecture.

It is probable, that the calx of antimony and phosphorated lime combine with each other. 1. Because it requires the application of heat and air for a shorter space of time to separate the sulphur from a given quantity of antimony mixed with bone-ashes than to produce this effect on antimony *by itself*: nor can the speedy calcination of antimony with bone-ashes be explained by supposing that the antimony can then bear more heat without melting; for the difference in the degree of heat applied in the two cases is not, apparently, sufficient to account for the difference of the times required for desulphurating the antimony. 2. Because it appears, that heat, applied to antimony in a considerable variety of degrees, and air for various spaces of time, formed a calx very different in colour, fusibility, and other chemical qualities, from that produced by calcining this metallic substance with bone-ashes. The strongest confirmation, perhaps, of the opinion that the antimonial calx and phosphorated lime are chemically united together is, that, however long the calcination of the antimony and bone-ashes is continued in the open vessel it will only produce precisely the same substance, with respect to chemical properties, that is produced the moment the sulphureous fumes cease.

But why is a snow-white powder produced by exposing a mixture of calcined antimony and bone-ashes to air and fire for a due length of time, and then applying a greater degree of fire in close vessels, whereas no such white powder is formed by a mixture of any calx of antimony and bone-ashes, exposed to any degree of fire in close vessels, without previous exposure to fire and air? The reason may be, that in order that the calx should unite with the phosphorated lime, it must be calcined to one

certain degree; which is effected by exposure to air and fire with the bone-ashes when it can part or combine with air, so as to be reduced to that state in which it will be duly calcined for union with that substance, which could not happen in close vessels.

If it be objected, that this explanation does not account for the *whiteness* of this preparation, which is only produced by a white heat, and to which air is not necessary, the difficulty will be removed by considering that this whiteness may be induced without any chemical alteration effected by the fire: for, after the first calcination in the open vessel, it seems to act, principally, in the same way that it does in making grey coloured bone-ashes, or imperfectly burnt bone, of a snowy whiteness, namely, by totally destroying matter extraneous to the phosphoric selenite. Fire also, in many instances, alters the colour of bodies without occasioning any change in their composition; and, perhaps, the change of the light clay or cineritious powder, formed by the calcination of antimony and bone-ashes in open vessels, to a snowy-white substance by further exposure to fire, depends in part upon its increase of specific gravity or other mechanical effects of fire. A striking example of the power of fire to change the colour of bodies, by merely increasing their specific gravity, is afforded by the operation of quartation, in which process, the silver being parted, the gold is left of the colour of copper; but, by exposure to a due degree of fire, it is changed to its well known yellow colour, without undergoing any alteration except an increase of specific gravity.

To elucidate the nature of the insoluble and infusible part of JAMES's Powder, I made the following experiments, in which I particularly had in view to determine whether several antimonial calces be wholly soluble in acids.

EXP. IX. (a) Needle-like crystals of Algaroth powder dissolved readily and totally in about thirty times their weight of marine acid.

(b) Part of the same parcel of crystallized Algaroth powder was calcined for above two hours, during which time it was exposed to as great a heat as it would bear without melting, and during which time it was constantly raked about. Nearly half of this calcined calx readily dissolved in marine acid, and by boiling the remainder in a proportionally much greater quantity of the same acid, great part of it was dissolved, and the small part which still resisted solution could not be dissolved in above 100 times its quantity of hot *aqua regia*. This indissoluble part afforded regulus with tartar by means of heat applied with the blow-pipe.

(c) White flowers of antimony generally left a residuum that was either insoluble, or dissolved with great difficulty, and in a small proportion, in marine acid or *aqua regia*; yet this residuum was reducible. Some parcels of this calx *totally* dissolved.

(d) A little of the antimony, long calcined in a former experiment, and afterwards melted into a yellow mass, Exp. 8. (a), would only partially dissolve in marine acid and *aqua regia*; but the copious residuum it left was reduced.

(e) Equal weights of crystals of Algaroth powder and calcined bone mixed together, dissolved *totally* and readily in marine acid. This shews, that disengaged phosphoric acid does not precipitate antimonial calx when marine acid is present.

(f) The *calx antimonii nitrata* of the Edinburgh Dispensatory, argentine flowers of antimony, hyacinthine glass of antimony, and calx precipitated from antimonial tartar by alkali of tartar, all dissolved readily and *wholly* in marine acid; but,

(g) Diaphoretic antimony left a residuum which mixed with tartar formed metallic grains under the flame applied by means of the blow-pipe.

(b) Any of the above soluble antimonial calces by further calcination with air and fire become more difficultly soluble, or partly indissoluble.

The next experiments were made principally for the purpose of knowing whether antimony calcined with vitriolic selenite, calcareous earth, and filiceous earth, would afford the same sort of calx as antimony calcined with bone-ashes.

EXP. X. 1500 grains of well burnt and dry plaster of Paris, mixed with as much pulverized antimony, were calcined together in the same manner as the mixture for making LILE'S Powder, Exp. 3. In half an hour the sulphureous fumes disappeared; after calcining half an hour longer in a heat that kept the bottom of the dish red hot, the mixture was of a reddish brown or copper colour, and after cooling weighed 2520 grains. Supposing, therefore, the whole deficiency of weight in this experiment to be from the sulphur carried off; and supposing the quantity of air combined with the metal to be the same as in Exp. 2. the loss of weight viz. 32 *per cent.* is more than would have been expected; but as in experiments of this nature it is not perhaps possible to repeat them under precisely the same circumstances, the difference of 2½ *per cent.* deficiency more than would have been calculated, may more reasonably be ascribed to the sublimation of antimony than to other causes. By exposure to 70° of fire in a close crucible, this calcined mixture changed to a pale straw-coloured powder, and the sides of the vessel were glazed yellow. The change of colour was the same in an open vessel in 60° of fire.

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Though it is probable, from this experiment, that there is an affinity between antimonial calx and vitriolic selenite, it is plain that the compound is very different from JAMES's Powder.

The next experiment with chalk and antimony, which Dr. BLAGDEN had the goodness to suggest, would lead to several conclusions, but I shall only take notice of the composition produced.

EXP. XI. 1200 grains of antimony were mixed with 800 grains of well washed, dried, and pulverized chalk, and calcined as in making LILE's Powder. In less than an hour the smell of sulphur disappeared; after which the mixture was calcined half an hour longer. It afforded a lighter clay-coloured powder than the calcination of antimony with bone-ashes; and weighed 1800 grains. By exposure to  $100^{\circ}$  of fire this powder changed to a dirty white colour. On examination, instead of aerated lime or chalk, there was found vitriolic selenite, part of which was probably combined with the antimonial calx; for, by means of boiling water repeatedly applied till the lixivium did not become turbid with muriated barytes nor acid of sugar, I could only obtain 12 *per cent.* of vitriolic selenite, mixed with a little antimonial calx; but by means of nitrous acid I separated 45 *per cent.* of this selenite, with scarcely any antimonial calx in it. The residuum, after this solution in nitrous acid, was calx of antimony with a little vitriolic selenite seemingly vitrified. Accordingly the composition may be stated to consist of 1000 parts of antimonial calx and 950 parts of vitriolic selenite which I infer from the quantity of selenite dissolved by the nitrous acid, and estimated to remain united to the calx; and from the following calculation of the proportion of these two ingredients formed in the experiment.

Antimony.	Sulphur.	Air.	
1200	- 300	+ 100	= 1000 Antimonial calx.

Calcar.	Aerial	Vitriolic	
earth.	acid.	acid.	
800	- 300	+ 450	= 950 Vitriolic selenite.

	1950
Loss by sublimation and waste	150
	<hr/> 1800

With regard to the nature of this calx, the greatest part of it readily dissolved in marine acid; and part of what then remained was also dissolved, but with great difficulty and very sparingly; a minute quantity resisted solution entirely.

EXP. XII. 600 grains of coarsely powdered antimony were mixed with 400 grains of purified white sand, and calcined as in making LILE's Powder. The smell of sulphur continued for one hour and a half, and the mixture was calcined for half an hour longer. On cooling, a brown powder was obtained which weighed 820 grains, and exposed to 100° of fire, melted into an irregularly figured, blackish mass, full of cavities.

In this experiment the loss of weight corresponds nearly to that in experiments above related, *viz.* those in which the deficiency of weight after calcining antimony alone was about 29½ *per cent.* The much longer time required in this experiment for carrying off the sulphur than in the calcinations with bone-ashes, gypsum, and chalk, perhaps is owing to there being no affinity between antimonial calx and siliceous earth.

I beg leave to mention one more experiment relative to JAMES's Powder.

EXP. XIII. A medicine is sold by F. NEWBERRY, under the title of "JAMES's Powder for Horses, Horned Cattle, Hounds, &c." It is a light clay-coloured, gritty, tasteless substance, in which  
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are seen small *spicula*. It appears to me to be nothing more than JAMES's Powder for Fevers, or LILE's Powder above-mentioned, made by calcining antimony and bone-ashes together in open vessels; because, 1st, by exposure to a white heat in close vessels, it turns as white as JAMES's Powder. 2dly, It dissolves partially in nitrous acid; and the remainder dissolves partially in marine acid. The nitrous solution contains phosphoric acid and calcareous earth; and the muriatic solution affords Algaroth powder.

From the whole of the above *analytical experiments* it appears :

1. That JAMES's Powder consists of phosphoric acid, lime, and antimonial calx; with a minute quantity of calx of iron, which is considered to be an accidental substance.

2. That either, these three essential ingredients are united with each other, forming a triple compound, or, phosphorated lime is combined with the antimonial calx, composing a double compound in the proportion of about 57 parts of calx and 43 parts of phosphorated lime.

3. That this antimonial calx is different from any other known calx of antimony in several of its chemical qualities. About three-fourths of it are soluble in marine acid, and afford Algaroth powder; and the remainder is not soluble in this menstruum, and is apparently vitrified.

From the preceding *synthetic experiments* it appears, that by calcining together bone-ashes, that is, phosphorated lime, and antimony in a certain proportion, and afterwards exposing the mixture to a white heat, a compound was formed consisting of antimonial calx and phosphorated lime, in the same proportion, and possessing the same kind of chemical properties, as JAMES's Powder.

